Green Synthesis of Nanoparticles from WPCBs

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Waste printed circuit boards (WPCBs), constituting approximately 10% of all electronic waste (e-waste), are particularly intriguing due to their high content of valuable metals and rare earth elements.

recycling WPCBs green chemistry e-waste nanoparticles

1. Significance of Waste Printed Circuit Boards (WPCBs)

E-waste, or electronic waste, is a rising global environmental concern due to the ever-accelerating accumulation of discarded electronic devices. In 2019, the global production of e-waste reached 53.6 million metric tons, and projections indicate a surge to 74.7 million metric tons by the year 2030 [1]. With electronic waste production currently estimated at around 52.2 million tons annually and a growth rate of 5%, the importance of recycling becomes increasingly evident [2].

Electronic waste includes a variety of components, with waste printed circuit boards (WPCBs) representing about 10% of total e-waste. WPCBs are of particular interest due to their high content of valuable metals like copper (Cu) and tin (Sn); precious metals such as silver (Ag), gold (Au), and palladium (Pd) [3]; and rare earth elements such as praseodymium (Pr), neodymium (Nd), lanthanum (La), and cerium (Ce), if compared with mineral ores [4]. The presence of these metals make electronic waste a potential resource for urban metal mining, reducing the need for virgin ore extraction [2]. However, it is crucial to acknowledge that e-waste also harbors hazardous elements like cadmium (Cd), mercury (Hg), nickel (Ni), and lead (Pb). Inadequate handling of these dangerous substances presents significant health and environmental risks [1].

Recycling WPCBs is imperative, for a triad of compelling reasons, namely environmental stewardship, resource conservation, and the promotion of circularity. From an environmental standpoint, the responsible recycling of WPCBs mitigates the hazardous impact associated with improper disposal, preventing the release of toxic elements into the environment. Embracing a circular economy, WPCB recycling promotes the reuse of extracted materials in manufacturing processes, reducing the reliance on virgin resources and minimizing overall waste generation [5].

2. Composition of WPCBs

WPCBs represent around 3–5% of the total weight of electronic waste [6]. According to a significant amount of research, WPCBs are mostly composed of polymers, ceramics, and roughly, up to 50% metals. The content of Fe, Cu, and Al ranges from 5% to 20%; the content of Zn, Sn, Ni, Pb, and Cr ranges from 0.1% to 10%; and the content of Au, Ag, Cd, Ta, Ta, Ge, Ga, In, Mn, Pb, Pd, Ba, Ti, and Bi is less than or equal to 0.1% [6][7][8][9][10]. **Table 1** shows the main composition of metals in WPCBs.

Table 1. Typical composition of primary metals found in WPCBs, according to literature.

Classification	Percent (%)	Element
Major elements	5–20	Fe, Cu, Al
Intermediate elements	0.1–10	Zn, Sn, Ni, Pb, Cr
Minor elements	<0.1	Ag, Au, Ba, Bi, Cd, Ga, Ge, In, Mn, Pb, Pd, Ta, Ti

WPCBs contain various valuable metals such as copper, silver, gold, and palladium, some of which are found on pins used for conducting electricity and fixing elements [11]. Au could be found in contacts of the cable. Silver is mainly concentrated on the pins (metal foil contacts) of the electronic components, and its highest content was found in the microchips. Other connector pins are made from Cu–Sn, Cu–Zn, or Cu–Sn–Zn alloys, which are covered with thin Ni or Au layers [7], making PCBs economically attractive for recycling.

In the process of nanomaterial synthesis, the generation of substantial chemical waste has raised massive environmental pollution concerns. While conventional techniques to mitigate the release of impurities, including chemicals and heavy metals, into the environment have been improved, opportunities for improvement in their efficiency, especially in dealing with smaller particles, remain [12]. As a result, ensuring the sustainability of synthesis methods becomes imperative, not only to curtail waste production, but also to optimize the efficiency of both nanomaterial production and waste treatment processes [13].

Microorganisms, plants, animals, and their derivatives, such as amino acids, enzymes, and organic acids, serve as natural sources for facilitating the green synthesis of nanoparticles. Green synthesis stands as a highly promising approach due to its versatility across various applications, offering numerous advantages over conventional methods [14].

The top-down or destructive method involves reducing bulk materials to nanoscale particles. Common techniques used for nanoparticle synthesis include mechanical milling, nanolithography, laser ablation, sputtering, and thermal decomposition. Unlike the bottom-up method, top-down synthesis is a slower and more expensive process [15], and it may not be suitable for large-scale production [16]. The conventional bottom-up method uses highly toxic substances, such as $NaBH_4$ and N_2H_4, as reducing agents [15], along with extreme temperatures [17] and specific pH conditions [6], and often requires high energy consumption. Few studies report the synthesis of nanoparticles via green routes using WPCBs as a substrate. Most studies use metallic salts, mainly nitrates and chlorides, as precursors. A summary of the most relevant green routes to produce nanoparticles from WPCBs is shown in **Figure 1**.

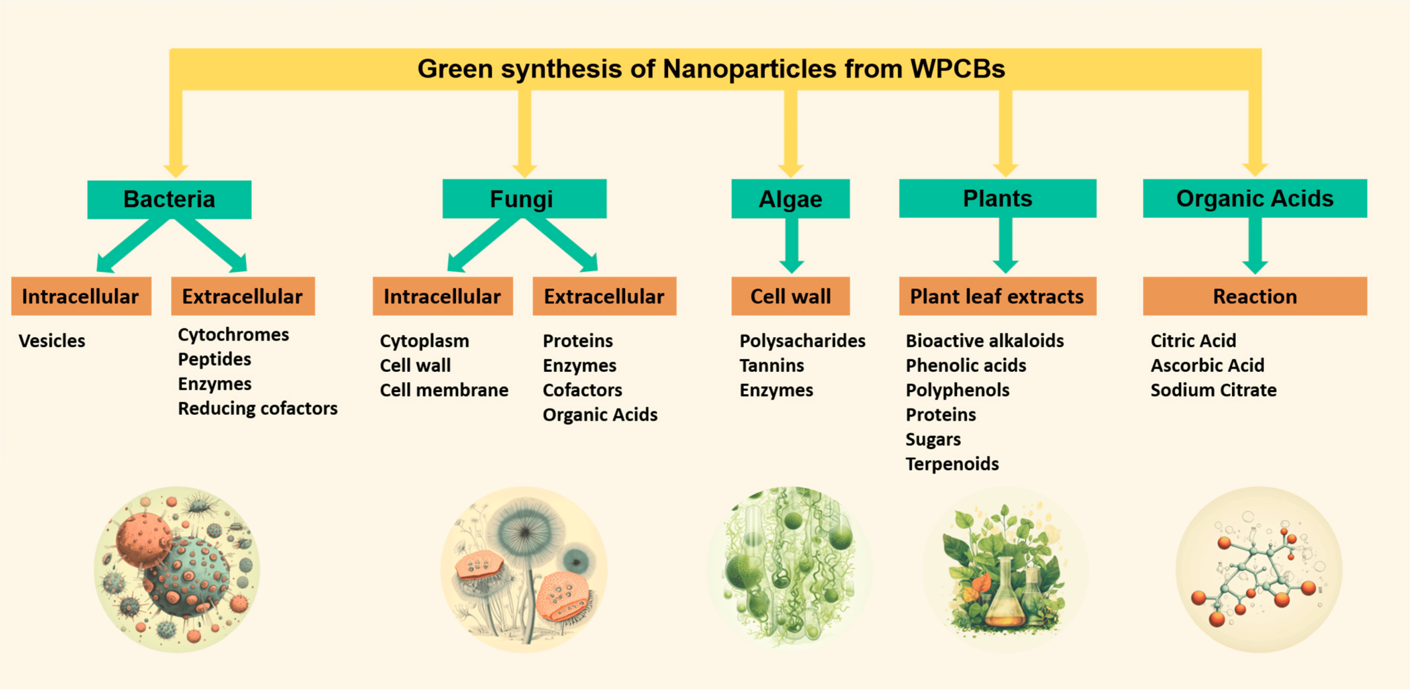

Figure 1. Relevant green routes for producing nanoparticles from WPCBs.

3. Green Organic Acids

The size and well-defined shapes of the nanoparticles depend, to a large extent, on the reducing agent and the action of a stabilizer in the synthesis of the solution phase.

The Turkevich method is a straightforward and reliable approach for synthesizing spherical particles ranging from 10 nm to 30 nm, using WPCBs as a substrate for Ag and Au [15]. The initial concentration of Ag was reported as 15.7 $g{\cdot}L^{-1}$. In this method, citrates are used as reducing agents [18][19].

A stable suspension of gold nanoparticles was achieved by utilizing a combination of sodium citrate and ascorbic acid as reducing agents, along with a stabilizing agent (polyvinylpyrrolidone—PVP), within a temperature range of 25–65 °C and a pH range of 2.5–4.0. This unique combination of reductive agents and a polymeric stabilizer enabled the production of pure gold metallic nanoparticles, featuring well-defined spheroidal and triangular shapes with a size distribution between 5–20 nm [4].

Ascorbic acid is commonly employed in the synthesis of nanorods. It serves the dual purpose of both reducing and stabilizing during the fabrication of copper nanoparticles (CuNPs) [20][21], silver nanoparticles (AgNPs) [15], and gold nanoparticles (AuNPs) [22]. Furthermore, it acts as an antioxidant, effectively reducing oxygen free radicals and metal ions. As a result, it is considered as a non-toxic reagent which can influence the growth, aggregation, and interaction of the synthesized particles with the external environment [20].

Glucose is another agent used to obtain NPs from WPCBs, and its effect is potentiated by ascorbic acid. In a study on the synthesis of CuNPs, this technique was used. The nanoparticles obtained exhibited round shapes, with sizes between 10–50 nm [23].

4. Plants

The use of plant leaf extract facilitates the biogenic reduction of metal ions into base metals. This process is characterized by its swiftness, as it occurs rapidly, and its ease of execution at room temperature and pressure. Furthermore, it can be conveniently scaled up. Synthesis mediated by plant extracts is environmentally friendly, and most methods used water [24] and alcohols, such as methanol and ethanol [25], to extract the main components from the plants. Plant extracts, which comprise bioactive compounds like alkaloids, phenolic acids, polyphenols, proteins, sugars, and terpenoids, are thought to play a crucial role in initially reducing metallic ions and subsequently stabilizing them [26]. For example, *Cathantharaus roseus* plant leaf extract was used to generate copper oxide nanoparticles from WPCB, as it was indicated that the amide groups present in the proteins and enzymes of leaf extract are responsible for the oxidation–reduction process, and that amine groups of the leaf extract act as capping agents. The particle size ranges were from around 5 nm–10 nm, exhibiting a polycrystalline nature [14].

In a ratio of 1:20 (solid/liquid), an aqueous extract of olive tree leaves was used as a source of polyphenols for the reduction of Cu, Cr, and Sn metals in WPCBS acidic leachates as a greener alternative for the recovery of valuable metals [24].

An aqueous extract of *Prosopis juliflora* leaves obtained by sonication, containing a pool of piperidine alkaloids, was used in equal proportions with PCB-derived copper nitrate at 80 °C to obtain copper oxide nanoparticles (gCON) for the oxidation–reduction process. The outcomes of optical, structural, and morphological analyses verified the existence of monocrystalline, spherical copper oxide nanoparticles, demonstrating an average size of 11 nm [27].

The leaf extract of *Cassia auriculata* was used as a reducing, as well as capping, agent in the synthesis of copper nanoparticles. The findings from this investigation have substantiated the potential of *Cassia auriculata* leaf extract for the recovery of copper from printed circuit boards, yielding nanoparticles with a size range of 50–100 nm. The CuNPs exhibited a prominent peak at 300 nm in UV–Vis spectra [28].

Aqueous extracts derived from aloe vera and geranium (*Pelargonium graveolens*) were employed to reduce copper ions present in the leaching solution, a product of copper shale (Kupferschiefier) bioleaching via chemolithotrophic bacteria, including *Acidithiobacillus ferrooxidans*. The utilization of *Pelargonium graveolens* extract resulted in the formation of copper nanoparticles (CuNPs) with a spherical shape and a size distribution ranging between 100 and 300 nm [29].

5. Bacteria

The bacterial synthesis of nanoparticles can occur through extracellular and intracellular processes, with various metals being reported for nanoparticle formation using different bacterial components such as biomass, supernatant, cell-free extracts, and their derivatives. Extracellular synthesis is generally preferred due to the ease with which nanoparticles can be recovered [30][31].

Cytochromes, peptides, cellular enzymes like nitrate reductase, and reducing cofactors all play crucial roles in nanoparticle synthesis within bacteria. Organic materials released by bacteria serve as natural capping and stabilizing agents for metal nanoparticles, preventing aggregation and ensuring long-term stability [32]. Consequently, bacteria are recognized as potential biofactories for synthesizing a wide range of nanoparticles, including gold, silver, platinum, copper, nickel, iron, palladium, titanium, titanium dioxide, magnetite, cadmium sulfide, etc. Given the toxicity of many metal ions to bacteria, the bioreduction of ions or the formation of water-insoluble complexes represents a defense mechanism developed by bacteria to mitigate this toxicity [33].

Intracellular synthesis has been observed, as demonstrated in CuNP synthesis by a bacterium isolated from the marine sponge *Hymeniacidon heliophila*. This bacterium exhibited an affinity for crucial metals released from waste printed circuit boards (WPCBs). Notably, at 30 °C, the bacteria secreted substances beneficial for copper bioleaching, while at 40 °C, metallic nanoparticles formed within the cells. This mechanism is believed to neutralize heavy metal toxicity by reducing the ionic force of metals, encapsulating metallic nanoparticles within the vesicles for later release. This transition from metal ions to non-toxic forms is considered to be a survival mechanism in contaminated environments [34].

Over a three-day period, *Cupriavidus metallidurans* and *Delftia acidovorans* synthesized gold nanoparticles from e-waste, with diameters ranging from 22 to 33 nm. *C. metallidurans* appears to counter gold toxicity through Au-regulated gene expression and the potential methylation of Au complexes, leading to the energy-dependent cellular accumulation of gold nanoparticles. In contrast, *D. acidovorans* employs a distinct gold precipitation mechanism involving the secretion of a secondary metabolite called delftibactin to protect itself from the toxic nature of Au^{3+}. In this case, gold nanoparticles are precipitated in the extracellular medium [35].

Finally, the management of waste generated in the plant leaf extract is mainly grouped into the following types: thermochemical treatments, such as combustion, gasification, and pyrolysis; biochemical treatments to obtain, for example bioethanol; drying methods; and the condensation of active components [36].

6. Fungi

Fungi play a crucial role in biogeochemical processes, capable of both dissolving and immobilizing metals. They have gained attention for their potential for synthesizing nanoparticles as part of biotechnological metal recycling from e-waste due to their remarkable resilience to high concentrations of heavy metals [37]. In contrast, bacterial fermentation processes often entail multiple steps to obtain a clear colloidal, including filtration, solvent extraction, and the use of sophisticated apparatuses that considerably increase the investment costs related to equipment [4].

The biosynthesis of metal nanoparticles using fungal cells can employ one of two mechanisms: (i) intracellular or (ii) extracellular synthesis routes. In intracellular synthesis, nanoparticles are formed and localized within the cytoplasm, cell wall, or cell membrane [38]. Initially, the metal ions, which serve as nanoparticle precursors, interact with oppositely charged cell surface components, where they can be simultaneously reduced to form nanoparticles, while remaining attached to the cell surface. These nanoparticles may then migrate to the cell membrane or cytoplasm. Alternatively, the ions may be internalized through active or passive transport mechanisms and subsequently reduced by intracellular reducing agents [39]. In the extracellular synthesis routes, fungal proteins, enzymes, cofactors, and metabolites like organic acids (e.g., citric acid, oxalic acid) play vital roles in the organism's survival and contribute to the reduction of metal ions into nanoparticulate forms. **Figure 2** provides a schematic representation illustrating the interaction between metals and the fungal cell surface.

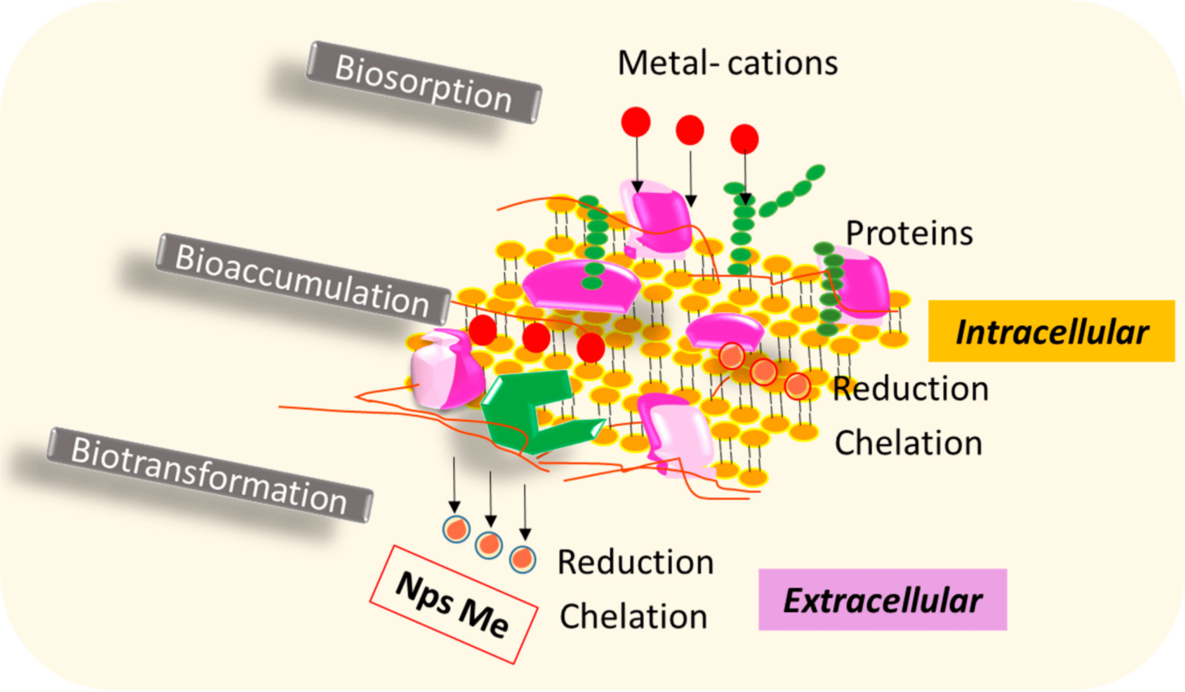

Figure 2. Biosynthesis of metal nanoparticles using fungal cells from e-waste.

Numerous groups of fungi are employed for the synthesis of nanoparticles from WPCBs, with *Fusarium oxysporum* emerging as a prominent organism capable of producing a variety of manufactured nanoparticles. The synthesis of silver nanoparticles (AgNPs) by *F. oxysporum* is primarily dependent on a reductase associated with NADH [4][40].

A mixture of *Fusarium oxysporum* and *Bacillus cereus* from e-waste synthesized Cu, Zn, Cd, and Au nanoparticles. The nanoparticles varied in size, but most of them possessed a diameter of less than 10 nm. They exhibited different shapes, including circular, triangular, and complex [41].

Trichoderma spp., *Aspergillus* spp., *Penicillium* spp., and *Verticillium* spp. are additional fungi that offer numerous advantages. These include diverse survival strategies, ease of biomass handling, the capacity to multiply using straightforward culture media, tolerance to high metal concentrations, and enhanced productivity in terms of nanoparticle production from WPCBs [42].

New research could be carried out, specifically using WPCBs as precursors and employing this fungal route for its multiple advantages in the synthesis of nanoparticles.

7. Algae

Algae represent another species known for its role in the bioreduction of metals, facilitating the production of gold and silver nanoparticles. This capability arises from the presence of specific functional groups, such as carboxyl, on their cell walls [12], along with the secretion of a polysaccharide called fucoidan, which aids in the intracellular synthesis of gold nanoparticles [22]. **Table 2** summarizes the relevant studies regarding nanoparticle reduction via the use of algae.

Table 2. Studies for nanoparticle reduction employing different algae.

Algae	Highlights	Element	Nanoparticle Size	Reference
Sargassum spp.	The microwave-assisted synthe-sis (MAS) of AgNPs using Sar-gassum spp. biomass involves the reduction of Ag+ ions by diverse organic compounds present in the macroalgae extract, including polysaccharides, proteins, poly-phenols, and flavonoids, through redox reactions.	Ag	10 to 175 nm. Average size of 36.43 nm	[43]
Rhizoclonium hieroglyphicum, *Lyngbya majuscule* and *Spirulina subsalsa*	The reduction of gold particles is attributed to the presence of cellular reductases, with proteins such as cysteine serving to stabilize the nanoparticles.	Au	<20 nm	[44]

Algae	Highlights	Element	Nanoparticle Size	Reference
Chlorella vulgaris	Carboxylate groups on the surface of *Chlorella vulgaris* cells capture metal ions, which are subsequently reduced to silver nanoparticles by reductase enzymes.	Ag	Average size 10.95 nm	[45]

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the pH conditions, this process results in monodispersed SNPs at low and neutral pH levels and nanorod structures in alkaline conditions [45].

Other algal species, including *Rhizoclonium hieroglyphicum*, *Lyngbya majuscule*, and *Spirulina subsalsa*, have also been identified for their capacity to visibly indicate gold reduction, specifically from Au(III) to Au(0), at both the intra- and extracellular levels. This is evidenced by a color change in their biomass to purple [44].

Green synthesis is presented as a sustainable alternative to traditional methods for the valorization of WPCBs. Within the green techniques mentioned here, some possess important aspects that are presented here as advantages; for example, obtaining nanoparticles using organic acids reduces synthesis times; however, in general, the stability of the nanoparticles obtained, along with their biocompatibility, is reduced. On the other hand, the use of microorganisms, such as bacteria and fungi, provide nanoparticles with defined and stable formats; however, the production cycles are broader, and there are limitations, including their inability to operate at high pulp densities, thus limiting their potential profitability. The use of plant extracts generates stable nanoparticles, but studies report the proportion of the plant extract and the chemical solution as the primary factor affecting the size of the NPs, along with their stability. Impurities of the extracts, limitations in process engineering, and operation stability are considered significant issues. Algae have demonstrated efficiency in the synthesis and stability in regards to the nanoparticles generated, but they present limitations concerning variability in the synthesis processes.

Finally, most of the waste generated in green metal extraction processes, as well as that derived from the synthesis of NPs, can be treated, or valorized, using three major processes, i.e., thermochemical, biological, and mechanical. The thermochemical type includes pyrolysis, gasification, and combustion processes, among others. The biological processes include fermentation, digestion, and the production of enzymes, and finally, the mechanical processes refer to operations such as drying, grinding, and pelletizing, in which added value can be provided to the waste generated in the green metal extraction processes. However, thermochemical processes remain the most required, due to their reduction of waste volumes and their recovery of molten metals [46].

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